

**THERMAL SHRINKAGE AND GAS VOIDS FORMATION OF  
WAXY CRUDE OIL**

By

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Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)

(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

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Jaykanesh Shamugam

A project dissertation submitted to the

Mechanical Engineering Programme

Universiti Teknologi PETRONAS

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BACHELOR OF ENGINEERING (Hons)

(MECHANICAL ENGINEERING)

Approved by,

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TRONOH, PERAK.

May 2013

## CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or person.

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JAYKANESH SHAMUGAM

## **ABSTRACT**

During the case of emergency or maintenance shutdown, crude oil in the pipelines undergoes thermal shrinkage and thus, high start-up pressure is needed to restart the crude flow. Research on thermal shrinkage and gas voids formation will therefore be helpful in identifying and maybe reducing the start-up pressure. The objectives of this project are to investigate the effect of cooling rate, Pour Point Depressant (PPD) and aging process on the gas void's volume, pressure and composition. The experiment was carried out by filling the crude oil inside a tube, heating it up and then cooling it. Few sets of experiments were carried out to analyze the effect of cooling rate, Pour Point Depressant (PPD) and aging process. The three parameters of the gas void; volume, pressure and composition were studied and recorded in the experiments. Based on the results, few conclusions were made. Firstly, the volume and pressure of the gas void increases with the cooling rate. Next, the presence of PPD in the crude oil slows down the thermal shrinkage and gas void formation process. Third, the aging process increases the volume and pressure of the gas void.

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# **CHAPTER 1**

## **INTRODUCTION**

### **1.1 BACKGROUND OF STUDY**

Petroleum is a naturally occurring flammable liquid which consist of a complex mixture of hydrocarbons <sup>[1]</sup>. Petroleum is one of the main energy sources that have been widely used around the world today. In the oil and gas industry, about 20% of the petroleum reserves produced and pipelined is crude oil. Basically, crude oil is the unrefined petroleum composed of hydrocarbon deposits <sup>[2]</sup>.

During normal operation, the crude oil which is taken out from the reservoir will be transported to storage tank through pipeline without much complication. However, during non-operating period or maintenance shutdown, thermal shrinkage and gas voids formation take place resulting in waxy crude oil. During this period, the low seabed temperature will cause the crude to gel up or form wax at Wax Appearance Temperature (WAT). The gelled crude with a high pour point needs high start-up pressure in order to start the crude flow for the transporting purpose compared to the liquid crude oil.

Using the assumption that the gelled crude behaves as an incompressible high viscous fluid <sup>[3]</sup>, the facility such as pump with a superior horse power is then needed to transport the waxy crude oil. High horse power equipment means high Capital Expenditure (CAPEX) to spend for the drilling and production process. Therefore, the thermal shrinkage and gas voids formation need to be analyzed so that the start-up pressure can be reduced during the case of emergency or maintenance shutdown and thus the operational cost can be minimized.

## **1.2 PROBLEM STATEMENT**

### **1.2.1 Problem Identification**

Thermal shrinkage and gas voids formed during cooling increase the restart pressure and thus, affect the transportation process of the crude oil. Therefore, the study of thermal shrinkage and gas voids formation in the gelled crude requires further qualitative and quantitative analysis in order to determine the accurate restart pressure.

### **1.2.2 Significance of the Project**

Studying the thermal shrinkage of the gelled up crude oil would further enhance the prediction of the restart pressure thus, avoiding any unnecessary excess in spending (reduces CAPEX) and ensures good flow of crude oil.

## **1.3 OBJECTIVE AND SCOPE OF THE STUDY**

The main objective of this project is to study the thermal shrinkage and gas voids formation of waxy crude oil. There are some sub-objectives for this project and they are:

- To investigate the effect of cooling rate on the gas void's volume, pressure and composition.
- To investigate the effect of Pour Point Depressant (PPD) on the gas void's volume, pressure and composition.
- To study the aging process of the gas void with respect to its volume, pressure and composition.

As mentioned above, the scope of study includes the measurement of volume, pressure and the composition of the gas void under different conditions.

#### **1.4 RELEVANCY OF THE PROJECT**

This project focuses on the characteristics of the gas voids under different parameters. The effect of cooling rate, Pour Point Depressant (PPD), aging process and the diameter of the test tube on the gas voids will be analyzed. Since cost and profit are of the utmost importance in every industry especially in the Oil and Gas industry, the outcome from this study will give a lot of benefits to the industry in terms of profit and cost benefit.

#### **1.5 FEASIBILITY OF THE PROJECT**

The project requires facilities to simulate the real pipeline so that the thermal shrinkage and gas voids formation can be well observed. The laboratory equipments that will be used are:

1. Water bath for cooling purposes
2. Gas Chromatography machine to analyze the gas voids composition.
3. Custom-made bubble line flow rig to simulate the thermal shrinkage of waxy crude oil in the pipeline.

All the facilities and equipments that need to be used are in good condition. Moreover, based on supervisor's feedback and Gantt chart, the project can be completed in the given period of time or even faster.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 WAXY CRUDE OIL**

Crude oil is a complex mixture of different types of hydrocarbons including paraffin, asphaltene, aromatic and resin. Among these components, high molecular weight paraffin and asphaltene are typically responsible for production and transportation problems in subsea pipeline systems. Crude oil is called as waxy crude oil when its paraffin content is relatively enough, typically ranging from 1% to 50% of paraffin <sup>[4]</sup>. Wax molecules are dissolved in the crude oil at reservoir temperatures (70-150°C) and pressures (50-100MPa). However, as the crude oil flows through a subsea pipeline resting on the ocean floor at a temperature of 4°C, the temperature of crude oil eventually decreases below its cloud point temperature or wax appearance temperature (WAT) because of the heat losses to the surroundings. The solubility of wax decreases drastically as the temperature decreases and wax molecules start to precipitate out of the crude oil. Waxy crude oils are well known to have a very complex rheological behaviour. Above the WAT, they behave as a simple Newtonian fluid. As the temperature drops below the WAT, the viscosity starts to increase sharply and to be sensitive to mechanical constraints, in relation to the presence of paraffin crystals and a gel-like structure in the material (Cawkwell *et al.* 1989).

#### **2.2 THERMAL SHRINKAGE**

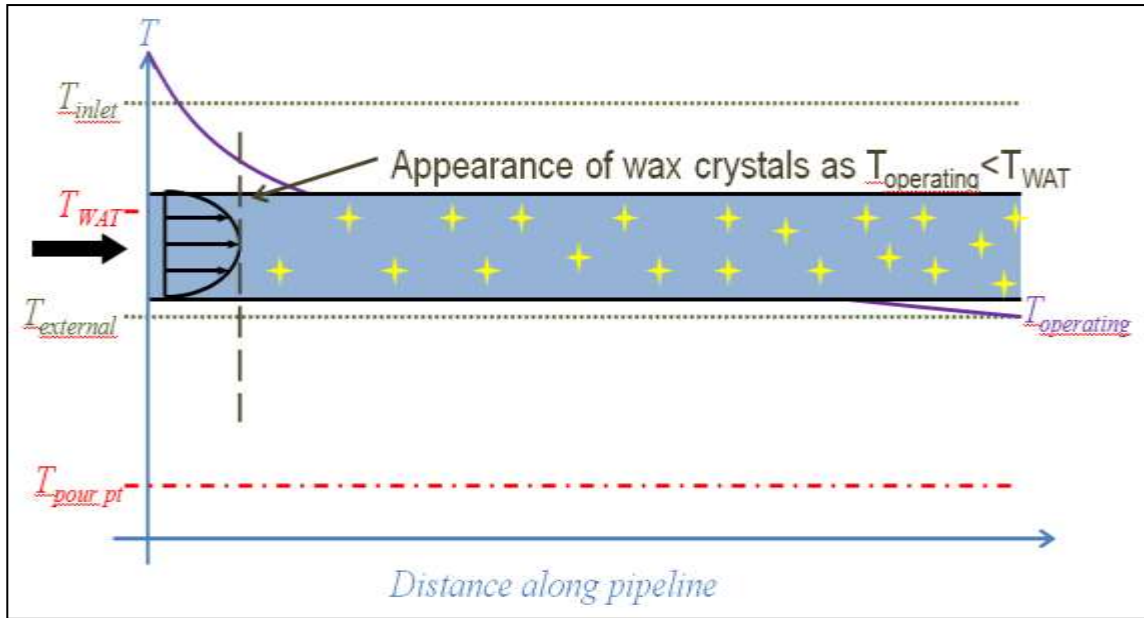
Basically, waxy crude oil undergoes thermal shrinkage when the pipeline temperature approaches the pour point. The pour point is the lowest temperature at which the crude

oil loses its flow characteristics. During the thermal shrinkage of the crude oil, wax or gelled crude will start to form at the Wax Appearance Temperature (WAT) and the wax starts to deposit at the pipeline forming a solid layer that decrease the available flow area. Major apprehensions arise during the restart of flow from the blocked pipeline due to the gelled up crude oil <sup>[5]</sup>.

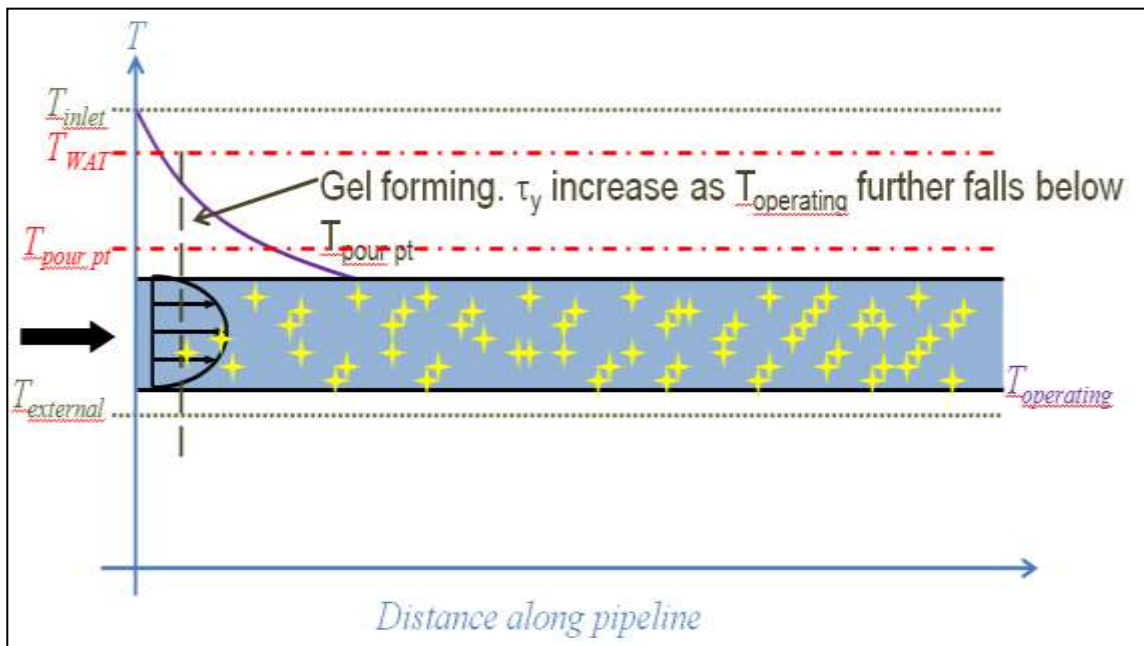
For flow to restart, a high pressure is needed in order to move the gelled crude compared to the crude oil that can easily flow in the pipeline using the assumption that gelled crude behaves as an incompressible high viscous fluid. The pressure required to break the gel and to restart the flow is proportional to the strength of the gel (yield stress) and the aspect ratio of the pipeline. Since high pressure is needed to move the gelled crude, a huge amount of money needs to be invested for the high horse power pump at the platform. A study on the thermal shrinkage entitled “Novel Approaches to Waxy Crude Restart: Part 1: Thermal shrinkage of waxy crude oil and the impact for pipeline restart” had came out with a result which proves that there were gas voids produced by cooling process of the crude oil in the flow line <sup>[6]</sup>. The gas voids appearance may affect the compressibility of the gelled crude since there are spaces for the gelled crude to move after some amount of pressure applied.



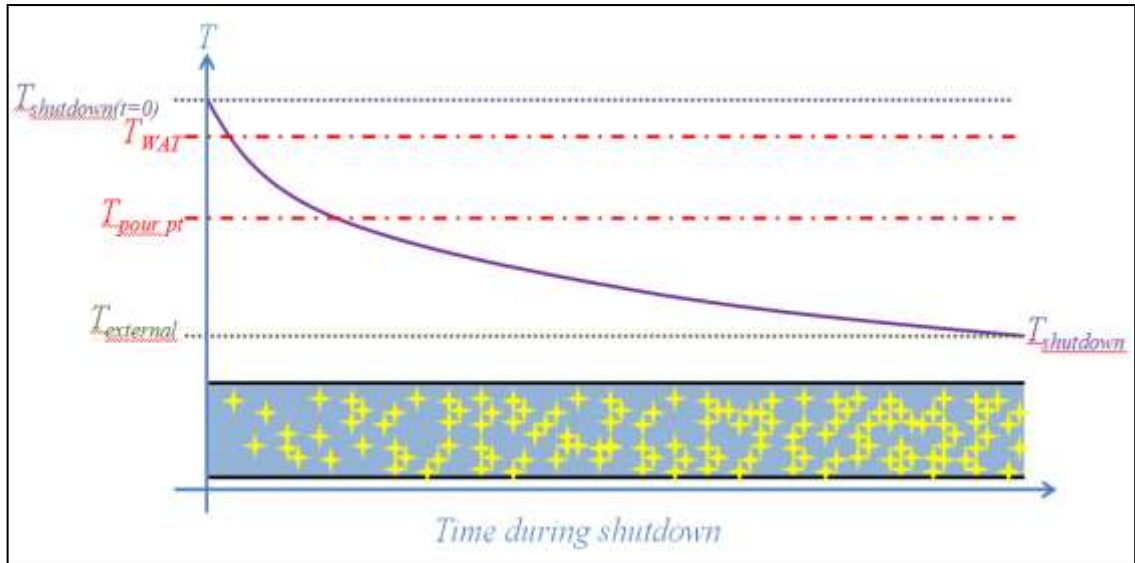
**Figure 2.1:** Cross section of paraffin deposition in a flow line (After Offshore Magazine, March 1997)



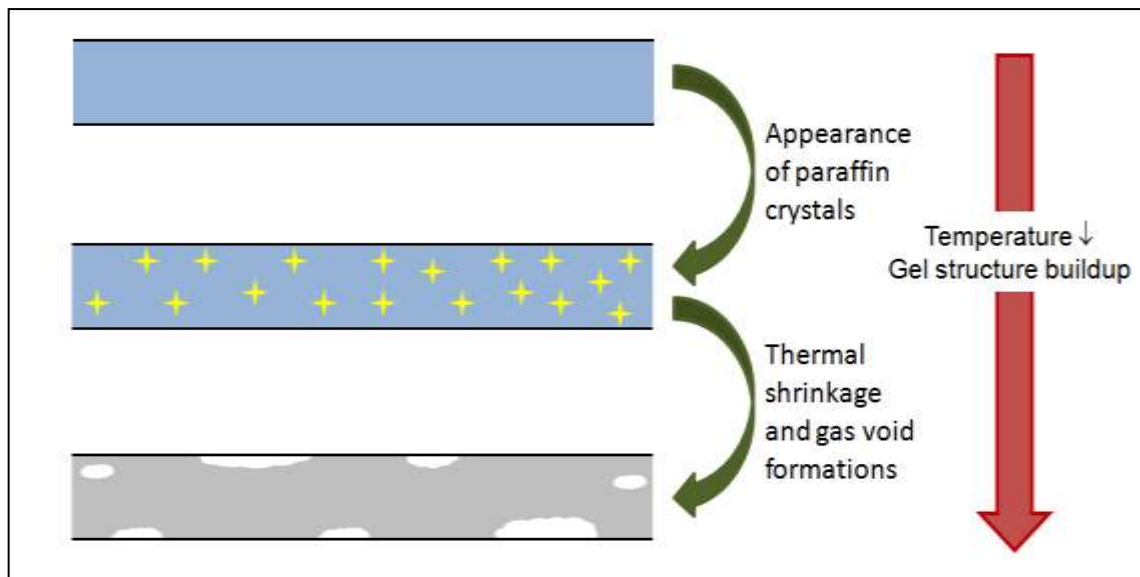
**Figure 2.2:** Wax formation start during thermal shrinkage



**Figure 2.3:** Wax form solid layer



**Figure 2.4:** Wax occupy in the pipeline



**Figure 2.5:** Wax formation stage



## **2.3 WAX APPEARANCE TEMPERATURE (WAT)**

The Wax Appearance Temperature (WAT) is also known as the cloud point temperature. WAT is the specific temperature at which wax develops within the liquid crude oil and when crystallization is visible <sup>[7]</sup>. The WAT point is determined by the concentration and molecular weight (properties) of the waxes and the chemical properties of the hydrocarbon matrix.

## **2.4 POUR POINT**

As stated by the ASTM standards (1983) <sup>[8]</sup>, the pour point is the temperature at which the flow of fluid ceases and the crude oil becomes a “frozen fluid” (Hansen et al., 1991; Claudy et al., 1988). Mustafa V. Kok et al. (1996) defined pour point as the point where a lattice leading to the solidification of the crude oil. As the amount of wax increases, crystals grow with the dropping temperature. When the crude is solidified (unable to flow) the point at which it happened is called the pour point temperature.

## **2.5 POUR POINT DEPRESSANT (PPD)**

Pour Point Depressant (PPD) is a chemical made of proprietary blend of specific polymers and surfactant. It is added to crude oil to lower the crude oil’s pour point. PPD alters the wax crystallization process, blocking the extensive growth of wax matrices in the crude oil. The change in wax crystalline structure and properties diminishes the ability of waxy aggregates to grow, thus resulting in lower pour point and improve the crude oil flow properties considerably along the production process or transportation pipeline. There are four basic types of PPD <sup>[9]</sup>, as listed below:

- Ethylene vinyl acetate copolymers
- Methacrylate copolymers
- Olefin/maleic anhydride copolymers
- Polysaccharides

## **CHAPTER 3**

### **METHODOLOGY**

#### **3.1 RESEARCH METHODOLOGY**

The following figure summarizes all of the steps involved during this study:



***Figure 3.1: Project Research Methodology***

### **3.2 PROCESS FLOW OF METHODOLOGY**

The detailed process flow of the project can be described as below:

- 1) First of all, the crude is heated up to 60°C for two hours for agitation process and to allow a good mixing between the heavy part and light part that are present in the crude.
- 2) The crude oil is then filled inside the tube and immersed in water bath to avoid thermal shock which can cause the crude to solidify once it is in contact with the tube. The mixture is then cooled from 60°C to 10°C.
- 3) The gas void formation is then observed for every 5°C interval. The condition of the crude oil inside the tube is recorded and picture of it is taken for further analysis. From this, the gas void's volume is measured.
- 4) After the final temperature is reached (10°C), the composition and pressure of the gas void are measured and analyzed. After two hours, the volume, pressure and composition are again measured and analyzed.
- 5) The experiment is repeated without the presence of Pour Point Depressant (PPD).

### 3.3 APPARATUS

- **Bubble Line Flow Rig**

1. Polyurethane tubing ID: 6mm (1 roll = 100meter).
2. PVC stopcock valve with compression fitting 8mm OD.
3. U-bend frame made of clear color acrylic sheet.

- **Equipment and Tools**

1. Water Bath Vivo RT2.
2. U-tube polyurethane – bubble line flow rig.
3. Syringe.
4. Gas Chromatography
5. Pressure Transmitter
6. Vernier Caliper
7. Magnetic Stirrer

### 3.4 PROJECT ACTIVITIES

The detailed activities of the project are as below:

#### 3.4.1 Fabrication

A new frame to hold the tube with crude oil is fabricated based on the previous frame. The new frame is fabricated keeping in mind that the tube must be fully immersed in the water bath so that the formation of the gas void at the apex is not affected.



*Figure 3.2: New frame setup*

### **3.4.2 Measurement of Gas Voids Pressure**

The measurement of gas voids pressure is done using pressure transmitter. The procedure is described as below:

1. The crude sample is heated at 60°C for 30 minutes to enable good mixing of the particles.
2. The crude sample is then filled inside the tube and the valve is closed.
3. Next, the sample is allowed to cool down to 10°C.
4. A syringe is connected to a pressure transmitter to record the pressure.
5. The syringe is then tapped at the apex of the tube where the gas voids are located due to pressure difference.
6. Few readings are taken to get the average pressure value.

### **3.4.3 Measurement of Gas Voids Volume**

The gas voids volume measurement can be done using Catia software. Below are the details on the method:

1. Before transferring the picture to Catia, two redlines are marked and the original parameter between the two redlines are measured.
2. Import picture to Catia software.
3. Draw the solid structure based on the sketch and determine the parameter scale.
4. Then, create another body with the exact void edge.
5. Extract the solid by Boolean and only left for the void body.
6. Lastly, measure the volume of the gas void solid and scale down to original ratio.

#### **3.4.4 Measurement of Gas Voids Composition**

Before analyzing the gas void composition using the Gas Chromatography machine, the gas void must be extracted from the tube without any contamination. Below are the details on the method that can be used to extract the gas void without any contamination.

1. Prepare two syringes, one empty and another one filled with helium.
2. Once the gas void is formed at the apex of the tube, tap both syringe and slowly release the helium gas and at the same time extract the gas void.
3. Suck the gas from the tube and seal it before transferring it to the Gas Chromatography machine.

#### **3.4.4 Wax Appearance Temperature (WAT) and Pour Point Determination**

Experiment was conducted to determine the Wax Appearance Temperature (WAT) and Pour Point of the Sepat-7 crude. The following steps illustrate how the experiment was conducted.

- 1) The crude sample is poured into the test tube and heated to 9°C above the expected pour point (36°C).
- 2) The crude sample is observed after the required temperature is reached.
- 3) The crude sample is then allowed to cool down to a temperature lesser by 3°C than the previous temperature. Once the required temperature is reached, the sample is observed again.
- 4) The crude sample is then continuously cooled down by a multiple of 3°C. The sample is observed at each temperature interval.
- 5) The temperatures at which wax starts to appear (WAT) and the crude sample ceases to flow completely (pour point) are recorded.

### 3.4.5 Preparation of Pour Point Depressant (PPD)

For this experiment, the concentration of PPD used is 500ppm.

$$\begin{aligned} 1. \quad V_{crude} &= \pi r_{tube}^2 L_{tube} \\ &= \pi (0.004^2)(0.2) \\ &= 1.0053 \times 10^{-5} m^3 \\ &= 10.053 \text{ mL} \end{aligned}$$

Where,

$$r_{tube} = 0.004m$$

$$L_{tube} = 0.2m$$

$$1m^3 = 1 \times 10^6 mL$$

2. Mass of crude

$$\begin{aligned} \% \text{ concentration } \left( \frac{w}{w} \right) &= \frac{ppm}{10,000} = \frac{X_{PPD}}{X_{PPD} + X_{crude}} \\ 0.05\% &= \frac{X_{PPD}}{X_{PPD} + X_{crude}} \end{aligned}$$

Where,

$$X_{crude} = \text{mass of crude}$$

$$X_{PPD} = \text{mass of PPD}$$

$$\text{Mass}_{\text{beaker}} = 50.077 \text{ g}$$

$$\text{Mass}_{\text{beaker} + \text{crude}} = 62.531 \text{ g}$$

$$\text{Mass}_{\text{crude}} = \text{Mass}_{\text{beaker} + \text{crude}} - \text{Mass}_{\text{beaker}} = 12.454 \text{ g}$$

$$\text{Volume}_{\text{crude}} = 10.053 \text{ mL}$$

2. Mass of PPD

$$0.05\% = \frac{X_{PPD}}{X_{PPD} + 12.454}$$

$$X_{PPD \text{ at } 10.053mL} = 0.6555g$$

Therefore, PPD with a mass of 0.655g will be used.

### 3.5 KEY MILESTONE AND GANTT CHART

#### Key Milestone

The following shows the key milestones of the project.

**Table 3.1: Key Milestone (FYP 1)**

<b>FYP 1</b>		
<b>No.</b>	<b>Detail</b>	<b>Week</b>
1	Submission of Extended Proposal	6
2	Proposal Defence	8
3	Submission of Interim Draft Report	13
4	Submission of Interim Report	14

**Table 3.2: Key Milestone (FYP 2)**

<b>FYP 2</b>		
<b>No.</b>	<b>Detail</b>	<b>Week</b>
1	Submission of Progress Report	8
2	Pre-SEDEX Poster & Presentation	11
3	Submission of Draft Report	12
4	Submission of Dissertation (soft bound) & Technical Paper	13
5	Oral Presentation	14
6	Submission of Dissertation (hard bound)	14



### Gantt Chart (FYP I)

Tasks/Weeks	3	4	5	6	7	8	9	10	11	12	13	14
Project Title Selection & Approval												
Literature review												
Crude Oil Sample Collection												
Wax Appearance Temperature (WAT) Determination												
Pour Point determination												
Crude Oil Sample Composition Determination												
Fabrication												
Initial Testing												
Determination of Best Method To Measure Gas Void												
Determination of Best Technique To Sample Gas Within Void												
GC Analysis												
Proposal Submission				22-Feb-13								
Proposal Defence						4-Mar-13	10-Mar-13					
Interim Report Submission												21-Apr-13

## Gantt Chart (FYP II)

Tasks/Weeks	3	4	5	6	7	8	9	10	11	12	13	14
Gas Void Analysis Without PPD												
Gas Void Analysis With PPD												
Gas Void Aging Process Analysis												
Gas Void Volume And Pressure Measurement												
Gas Void Composition Analysis Using GC												
Effect Of Cooling Rate Analysis												
Effect Of Tube Diameter Analysis												
Progress Report Submission												
Pre-SEDEX Poster & Presentation												
Draft Report Submission												
Dissertation (soft bound) & Technical Paper Submission												
Oral Presentation												
Dissertation (hard bound) Submission												

## CHAPTER 4

### RESULTS AND DISCUSSION

#### 4.1 POUR POINT ANALYSIS

Experiment was conducted to determine the Wax Appearance Temperature (WAT) and Pour Point of the Sepat-7 crude. The experiment was done using test tube and water bath and the results were tabulated.

*Table 4.1: Pour Point experiment result for Sepat-7*

Temperature (°C)	46	43	40	37	34	31	28	25
<b>Crude without PPD</b>	no changes observed	wax starts to appear	wax content is more visible	cease to flow completely	cease to flow completely	cease to flow completely	cease to flow completely	cease to flow completely
<b>Crude with PPD</b>	no changes observed	no changes observed	no changes observed	no changes observed	wax starts to appear	wax content is more visible	cease to flow completely	cease to flow completely

From the results, we can see that the wax starts to appear in the crude sample at the temperature of 43°C. At the temperature of 40°C, the wax content is more visible and starts to cover most of the crude. Finally, at 37°C, the crude ceases to flow completely. Therefore, based on the experiment, we can conclude that for Sepat-7 crude, the Wax Appearance Temperature (WAT) is 43°C and the Pour Point is 37°C. However, with the presence of PPD in the crude, the new WAT and Pour Point obtained are 34°C and 28°C. This shows that PPD has lowered both the WAT and Pour Point of the crude by 9°C.

## 4.2 EXPERIMENT: VARIATION OF FINAL TEMPERATURE (COOLING RATE)

### 4.2.1 Experiment 1



Condition:

Final temperature: 20°C

Time taken to cool down: 1hour 41 minutes

Results:

*Table 4.2: Experiment 1 result*

Temperature	Time	Condition
60°C	2.15pm	
20°C	3.56pm	

### 4.2.2 Experiment 2



Condition:

Final temperature: 10<sup>0</sup>C

Time taken to cool down: 2 hours 03 minutes

Results:




***Table 4.3: Experiment 2 result***




Temperature	Time	Condition
60 <sup>0</sup> C	10.17am	
10 <sup>0</sup> C	12.20pm	




#### 4.3 EXPERIMENT: THE FORMATION OF GAS VOID WITHOUT THE PRESENCE OF POUR POINT DEPRESSANT (PPD)

Results:



*Table 4.4: Experiment result*

Temperature	Condition
60 <sup>0</sup> C	No gas void detected 
55 <sup>0</sup> C	No gas void detected 
50 <sup>0</sup> C	No gas void detected 

45°C	<p>No gas void detected</p> 
40°C	<p>No gas void detected</p> 
35°C	<p>No gas void detected</p> 

30°C	<p>No gas void detected</p> 
25°C	<p>Gas void detected at the apex and left portion of the tube</p> 
20°C	<p>Gas void detected at the apex and left portion of the tube</p> 









15 <sup>0</sup> C	<p>The gas void starts to expand</p> 
10 <sup>0</sup> C	<p>The gas void continues to expand</p> 




#### 4.4 EXPERIMENT: THE FORMATION OF GAS VOID WITH THE PRESENCE OF POUR POINT DEPRESSANT (PPD)



Results:

*Table 4.5: Experiment result*

Temperature	Condition
60 <sup>0</sup> C	No gas void detected 
55 <sup>0</sup> C	No gas void detected 
50 <sup>0</sup> C	No gas void detected 

45°C	<p>No gas void detected</p> 
40°C	<p>No gas void detected</p> 
35°C	<p>No gas void detected</p> 




30°C	<p>No gas void detected</p> 
25°C	<p>Gas void detected at the apex of the tube</p> 
20°C	<p>Gas void detected at the apex and left portion of the tube</p> 




15 <sup>0</sup> C	<p>The gas void starts to expand</p> 
10 <sup>0</sup> C	<p>The gas void continues to expand</p> 


#### 4.5 EXPERIMENT: THE AGING PROCESS OF THE GAS VOID

Results:

*Table 4.6: Experiment result*

Temperature	Condition
60 <sup>0</sup> C	No gas void detected 
25 <sup>0</sup> C	Gas void detected at the apex of the tube 
20 <sup>0</sup> C	The gas void starts to expand 

<p>20°C (after 30 minutes)</p>	<p>Gas void detected along the tube</p> 
<p>15°C</p>	<p>Gas void detected along the tube</p> 
<p>15°C (after 30 minutes)</p>	<p>Gas void detected along the tube</p> 

10°C	<p>Gas void detected along the tube</p> 
10°C (after 30 minutes)	<p>Gas void detected along the tube</p> 



## 4.6 COOLING RATE

### Experiment 1

Final temperature: 20°C

Time taken to cool down: 1hour 41 minutes

*Table 4.7: Gas void volume and pressure*

Experiment	Volume (m3)	Pressure (kPa)
1	7.84E-07	-0.0521
2	7.88E-07	-0.0499
3	7.62E-07	-0.0487

$$\text{Cooling rate} = \frac{60^{\circ}\text{C} - 20^{\circ}\text{C}}{101 \text{ minutes}}$$

$$\text{Cooling rate} = 0.396 \text{ degree Celsius /minute}$$

### Experiment 2

Condition:

Final temperature: 10°C

Time taken to cool down: 2 hours 03 minutes

*Table 4.8: Gas void volume and pressure*

Experiment	Volume (m3)	Pressure (kPa)
1	1.31E-06	-0.0489
2	1.44E-06	-0.0477
3	1.28E-06	-0.0481

$$\text{Cooling rate} = \frac{60^{\circ}\text{C} - 10^{\circ}\text{C}}{123 \text{ minutes}}$$

$$\text{Cooling rate} = 0.407 \text{ degree Celsius /minute}$$

## 4.7 GAS VOID PRESSURE MEASUREMENT

### Without PPD

Temperature = 10°C

*Table 4.9: Gas void pressure reading without PPD*

Experiment	Pressure (kPa)
1	-0.0517
2	-0.0481
3	-0.0556

### With PPD

Temperature = 10°C

*Table 4.10: Gas void pressure reading with PPD*

Experiment	Pressure (kPa)
1	-0.0445
2	-0.0543
3	-0.0497

### During Aging Process

Aging period = 0 minute

Temperature = 10°C

*Table 4.11: Gas void pressure reading at 0 minute aging period*

Experiment	Pressure (kPa)
1	-0.0517
2	-0.0481
3	-0.0556

Aging period = 30 minutes

Temperature = 10°C

*Table 4.12: Gas void pressure reading at 30 minutes aging period*

Experiment	Pressure (kPa)
1	-0.0589
2	-0.0646
3	-0.0621

#### 4.8 GAS VOID VOLUME MEASUREMENT

##### Without PPD

Temperature (20°C)		Temperature (15°C)	
Experiment	Volume(m3)	Experiment	Volume (m3)
1	7.94E-07	1	1.08E-06
2	7.74E-07	2	1.21E-06
3	7.90E-07	3	1.29E-06

Temperature (10°C)	
Experiment	Volume (m3)
1	1.45E-06
2	1.59E-06
3	1.42E-06

**Table 4.13:** Gas voids volume at 20°C, 15 °C and 10°C (without PPD)

##### With PPD

Temperature (20°C)		Temperature (15°C)	
Experiment	Volume(m3)	Experiment	Volume (m3)
1	1.70E-08	1	2.15E-08
2	1.60E-08	2	2.25E-08
3	1.56E-08	3	2.11E-08

Temperature (10°C)	
Experiment	Volume (m3)
1	2.47E-08
2	2.53E-08
3	2.51E-08

**Table 4.14:** Gas voids volume at 20°C, 15 °C and 10°C (with PPD)

### Aging Process

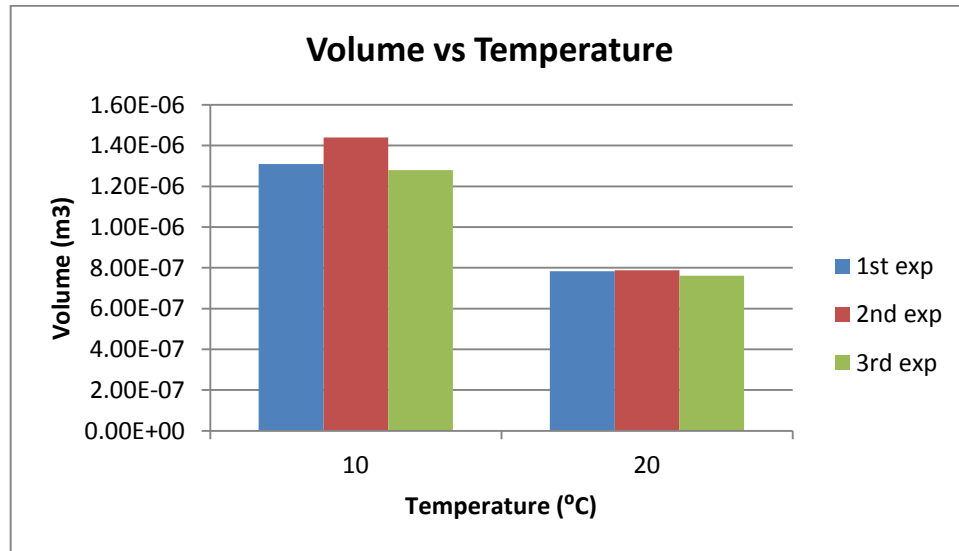
Temperature (10°C)	
Experiment	Volume(m3)
1	1.35E-06
2	1.49E-06
3	1.45E-06

Final Temperature, 10°C (after 30 minutes)	
Experiment	Volume(m3)
1	1.64E-06
2	1.67E-06
3	1.55E-06

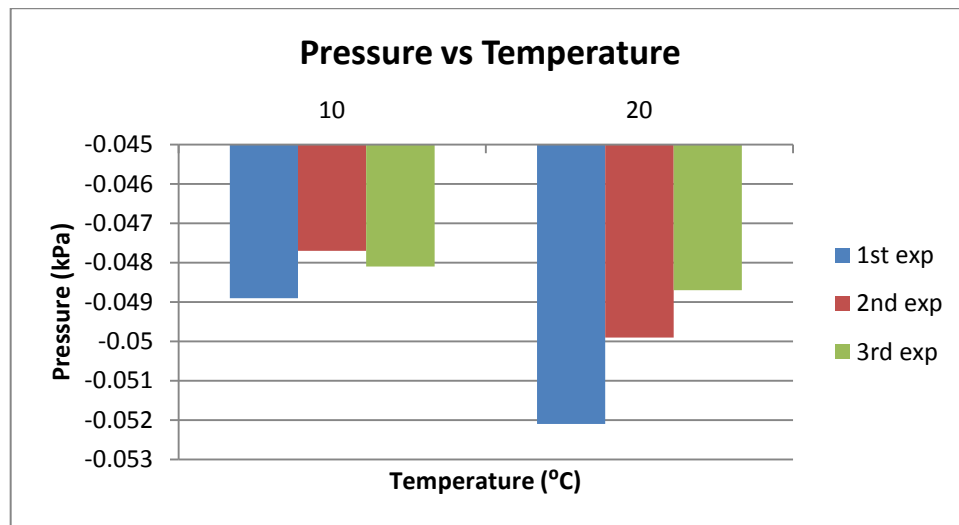
**Table 4.15:** Gas voids volume at 10°C

Based on the results obtained, graphs are constructed as follow:

#### Variation of Final Temperature (Cooling Rate)

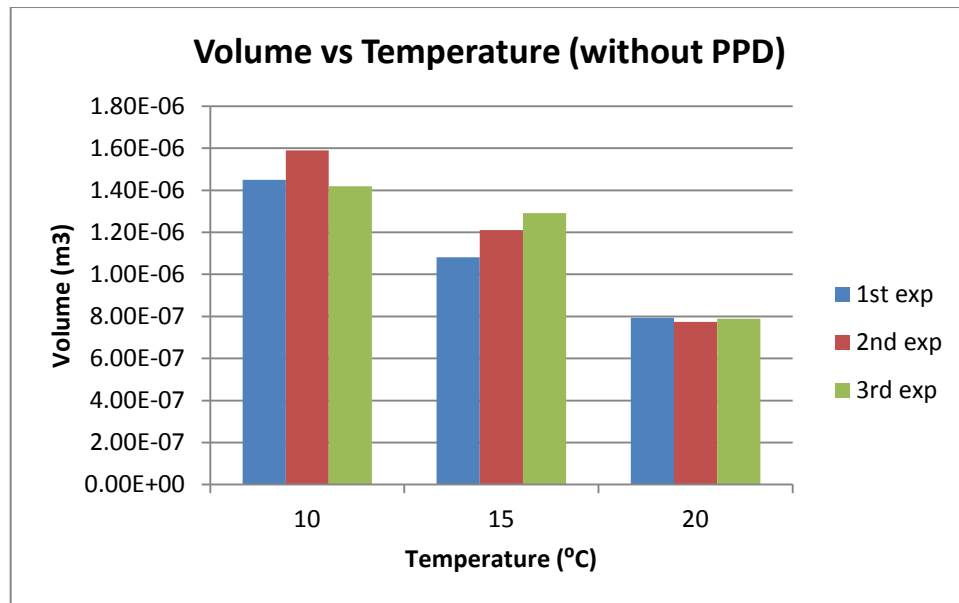


*Figure 4.1: Volume versus Temperature Graph*

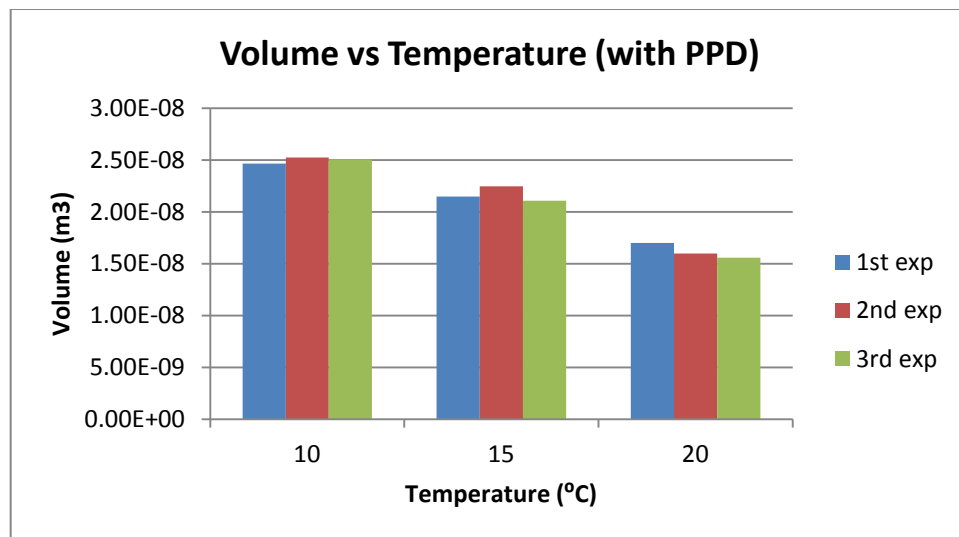


*Figure 4.2: Pressure versus Temperature Graph*

**The Formation Of Gas Void With And Without The Presence Of Pour Point  
Depressant (PPD)**

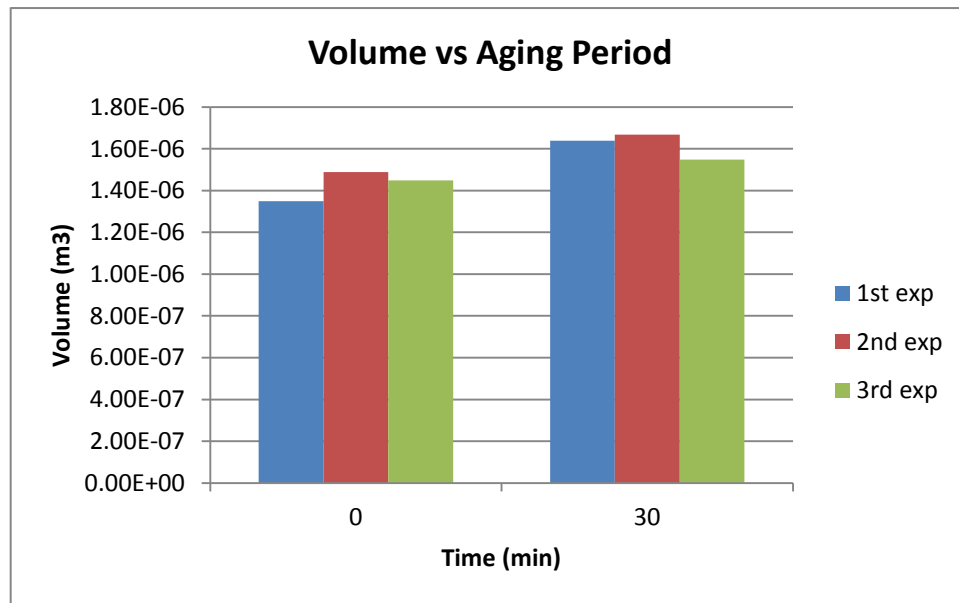


*Figure 4.3: Volume versus Temperature Graph*

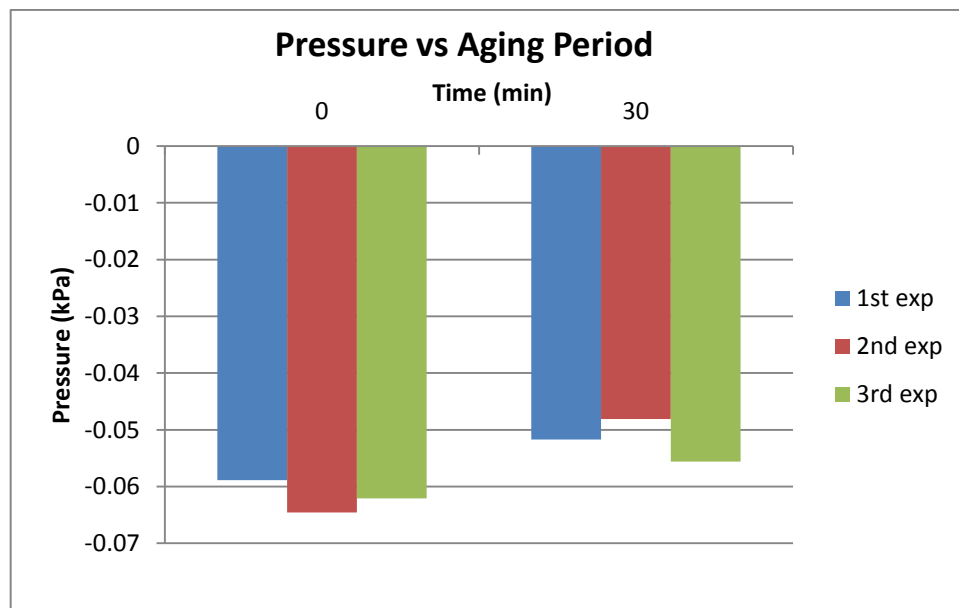


*Figure 4.4: Volume versus Temperature Graph*

## The Aging Process Of The Gas Void



*Figure 4.5: Volume versus Aging Period Graph*



*Figure 4.6: Pressure versus Aging Period Graph*

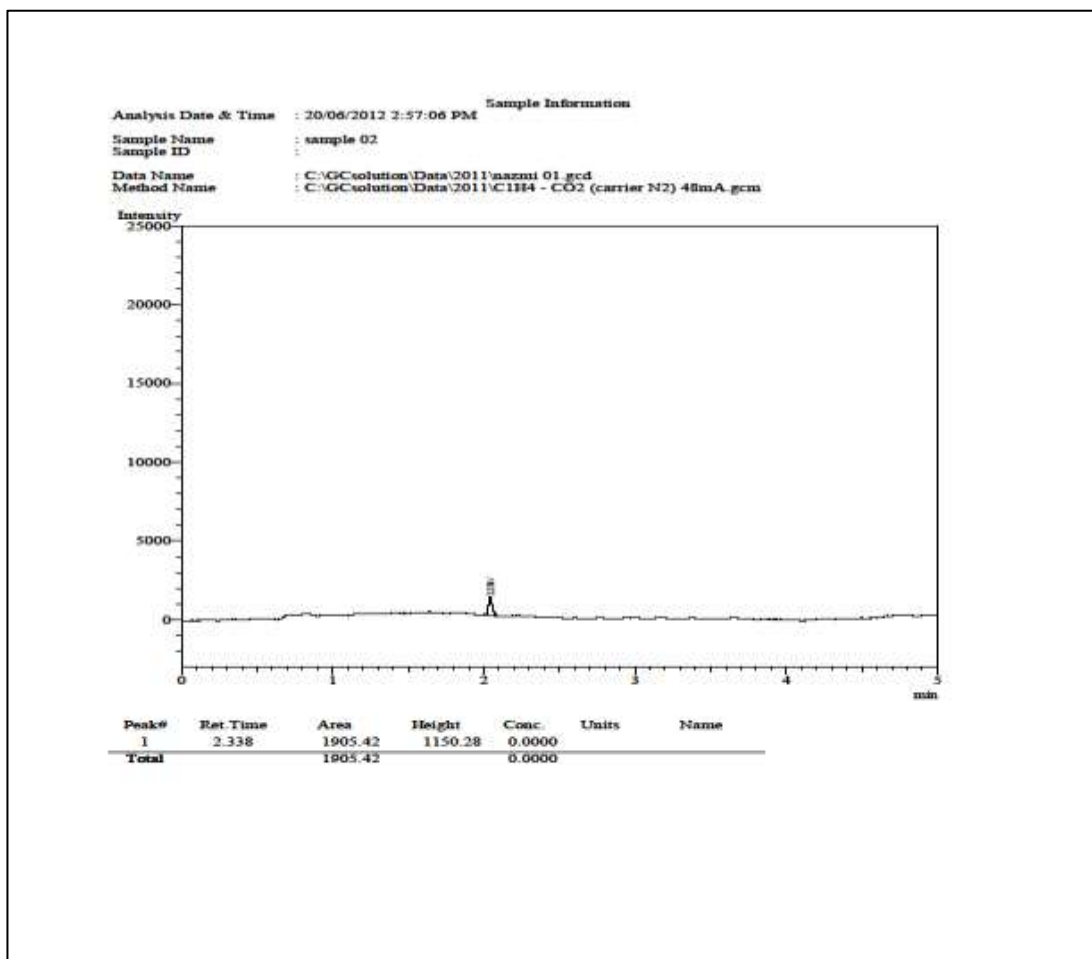
Based on the experiments done and the results obtained, few things can be concluded regarding the thermal shrinkage and gas void formation. From the variation of final temperature experiment that was conducted to investigate the effect of cooling rate on the gas void formation, it is found out that the volume and pressure of the gas void increases with the cooling rate. As the final temperature decreases and the time taken for cooling process increases, higher percentage of gas void is observed along the tube. This is because at lower temperature and higher cooling rate, the gas void is able to expand more and fills the tube. This proves that thermal shrinkage and gas void formation is enhanced by low temperature.

Besides, the effect of Pour Point Depressant (PPD) on thermal shrinkage gas void formation was also studied. Crude oil with and without the presence of PPD were cooled down to the temperature of 10°C. From the results, it can be seen that gas void starts to form at the apex of the tube at the temperature of 25°C (below the pour point of the crude) for both the experiments. However, the gas void tends to expand more in the crude oil without PPD and fills the tube as the temperature goes down. This shows that the presence of PPD does affect the gas void formation. When PPD is added to the crude oil and then cooled down, lower volume of gas void is formed compared to the crude oil cooled without the presence of PPD. This is because the addition of PPD helps to reduce the wax precipitation and lower the melting point of the wax. However, the addition of PPD does not stop the gas void formation process completely but rather slows it down.

Regarding the study on aging process, the result shows that the volume and pressure of the gas void increases slightly during the aging process. Based on the observation, the gas void expands and starts to fill up the tube during the aging process. This happens because the gas void formation is proportional to time. As the crude oil is allowed to cool down further, higher volume of gas void is observed.



## 4.9 GAS VOID COMPOSITION



*Figure 4.7: Gas Chromatography result*

The figure above shows the Gas Chromatography result obtained from the previous research. The graph shows that only one peak occurs. This is because the gas void formed contains only air. However, based on thorough research and study, the gas void should be composed of gases such as carbon dioxide, helium, hydrogen, nitrogen, methane, ethane, butane and propane with methane being in highest amount (70% - 98%). Thus, the graph should produce more than one peak. The result produced is not accurate because the gas void might be contaminated during the extraction process.

## **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

#### **5.1 CONCLUSION**

Thermal shrinkage and gas void formation take place when the waxy crude oil undergoes cooling process below its pour point. This project was carried out with few objectives in mind that are to investigate the effect of cooling rate, Pour Point Depressant (PPD), and aging process on the gas void's volume, pressure and composition. Based on the results obtained, few conclusions are made.

First of all, the volume and pressure of the gas void increases with the cooling rate. Next, the presence of PPD in the crude oil slows down the thermal shrinkage and gas void formation process. Although gas void starts to form in the crude at the temperature of 25°C with and without the presence of Pour Point Depressant (PPD), however, the gas void tends to expand more in the crude oil without PPD and fills the tube as the temperature goes down. On the other hand, the aging process increases the volume and pressure of the gas void. After the aging process, the gas voids are more visible and appears along the tube.

## **5.2 RECOMMENDATIONS**

Few improvements that can be done for future work are:

1. Use different types of crude oil to compare the characteristics.
2. Figure out some other new methods to measure the pressure, volume and composition of the gas void which could provide better results.
3. Use special syringe to extract the gas void so that the gas void would not be contaminated.
4. Study on other parameters besides volume, pressure and composition of the crude oil may provide a better understanding on the thermal shrinkage and gas void formation.
5. Study the structure of the wax formed using microscope to get a better picture.

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## APPENDICES

### Measurement of Gas Voids Volume Using Catia

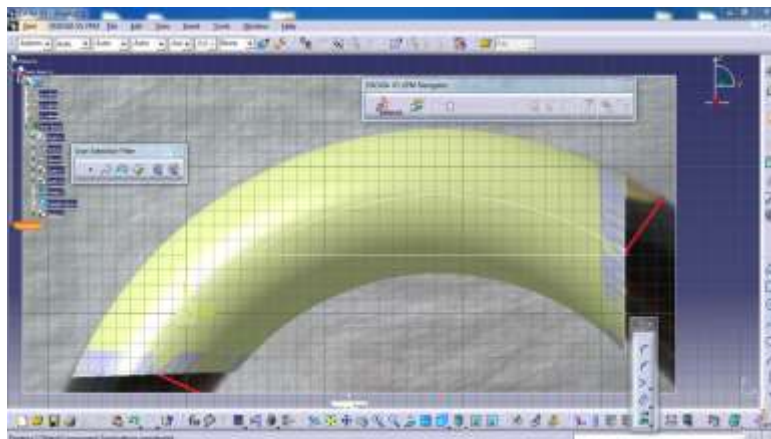
1. Before transferring the picture to Catia, two redlines must be mark and the original parameter between the two redlines must be measured.



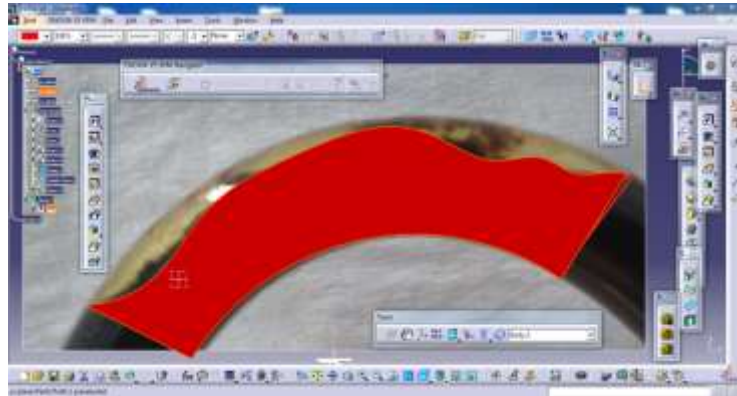
2. Import the picture into Catia.



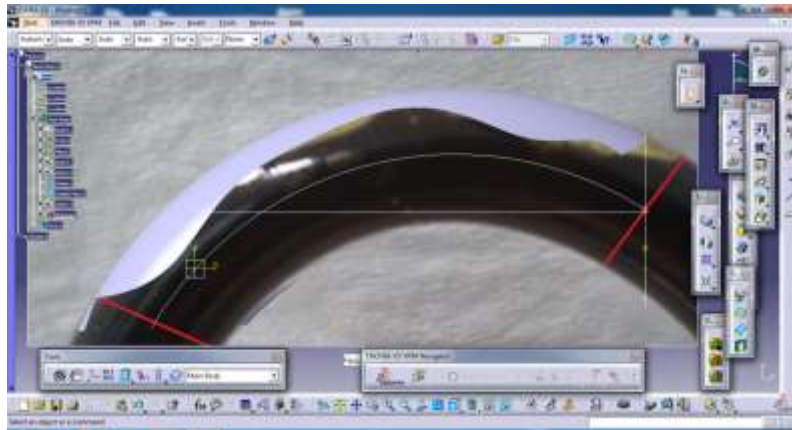
3. Draw the solid structure of the pipe based on the sketch and determine the parameter scale.



4. Then, create another body with the exact void edge.



5. Extract the solid by Boolean and only left for the void body.



6. Lastly, measure the volume of the gas void solid and scale down to original ratio.

